MICROANALYTICAL FACILITIES AT HP-HT LABORATORY AT ISTITUTO NAZIONALE DI GEOFISICA E VULCANOLOGIA, ROME (ITALY)

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Introduction

The usefulness of quantifying elemental compositions and observation of microstructures is invaluable in the sciences of mineralogy, petrology, structural geology and materials research. Morphology, texture and microstructure of minerals and rocks can be observed. The composition of several mineral phases as well as the composition of a rock-forming mineral relative to its growth history can be measured; for example, its core and rim (eventually different) compositions. Material science applications are even more varied, from thin film semi-conductors to photonics materials to super conductors. Computer power and image analysis software allow mapping of element distributions at various scales. Software applications are not only providing the absolute elemental concentration, but also the spatial distribution of elemental concentrations. New software and hardware allow elements to be qualitatively or quantitatively mapped, over almost any area on a sample.

Scanning electron microscopy (FESEM) or electron probe microanalysis (EPMA) are generally considered micro-analytical techniques which are able to *image* or *analyze* materials we can not generally observe with the resolution offered by visible techniques.

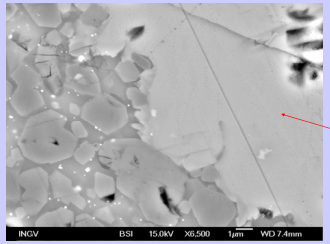
By *image* we mean photograph an object much smaller than we can see, even with the aid of an optical microscope. The FESEM generates much less electrostatically distorted *images* with spatial resolution lower then 2 nm, i.e. from 3 to 6 times higher resolution then conventional SEM. FESEM is equipped with detectors for x-ray (EDS) and for secondary and back scattered electrons and can increase probe current up to 200 nA improving signal to noise ratio in x-ray map. Moreover it is possible to collect **chemical maps** and *images* from a large area of sample through a special software (navigator). **Backscattered electrons** (BSE) are high energy electrons emitted from the specimen as a result of the high energy electron probe's interaction within the specimen. BSE emission is the result of elastic events between primary electrons and other electrons within the specimen which are relatively tightly bound. BSE emission intensity is very much a function of the specimen's atomic number; i.e., the higher the atomic number, the brighter the image.

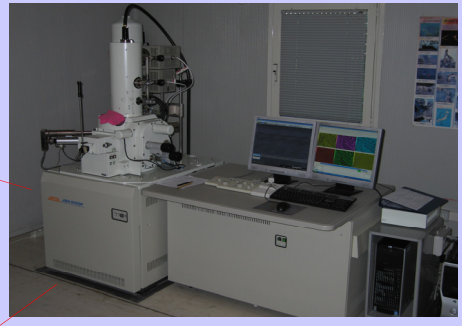
By *analyze* we mean identify the elements (e.g., silicon, iron, etc) of which the specimen is composed. Elemental analysis can also be accomplished at a micro-scale; for example, EPMA can probe a specimen as small as 5 thousands of a millimeter (5 microns), and not only identify the elements present but measure them with a small degree of error. These instruments represent two if the greatest advances in scientific instrumentation, however they do have their limits. For example, not all specimens can be exposed to the high vacuum within the specimen chamber. Also, elements lighter than atomic number 8 (oxygen) can not be measured without reservations, and EPMA is not sensitive to many elements below 100ppm. Still, this instrumentation has proved invaluable, especially for mineralogists and petrologists, and a good operator can vary one parameter or another to circumvent instrumental weaknesses. The EPMA is designed to measure qualitatively composition of a solid polished material on a microscale with high precision (less then percent relative for major constituents) and low detection limits (commonly a few tens to few hundreds ppm). Sample of interest can be as small as a few microns across.

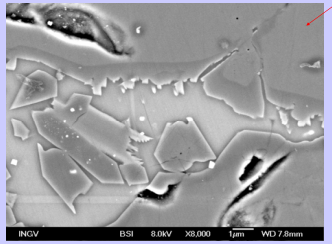
Technical information

The High Pressure High Temperature Laboratory of Experimental Volcanology and Geophysics acquired new microanalytical facilities in 2006, such as a Field Emission Electron Microscope (FESEM) JEOL JSM 6500 F and a Microprobe (EPMA) JEOL JXA 8200.
The FESEM, witch reach resolution better then 2 nm, is equipped with detectors for x-ray (EDS) and for secondary and backscattered electrons. Moreover it is possible to collect chemical maps and images from a large area of sample through a special software (Navigator).

The microprobe (EPMA) is WD/ED combined microanalyser. The combination of up to 5 wavelength dispersive X-ray spectrometers (WDS) and an energy dispersive X-ray spectrometer (EDS) assures the most efficient and accurate analysis. 12 analyzing crystals, allocated in the 5 spectrometer (4 in the first one and 2 in the other) cover a large range of analyzing elements.
Backscattered electron, secondary electron and X-ray imaging facilities are also available.
A transmission illuminator is used to observe thin specimen in transmitted light under the optical microscope.
A software (Specimen Navigator) allows to select point of analysis on scanned image of thin section of rock specimens.

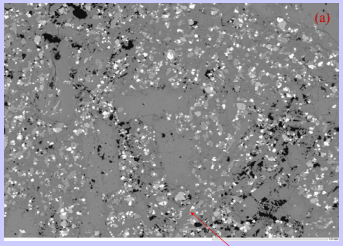


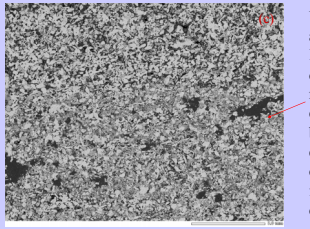




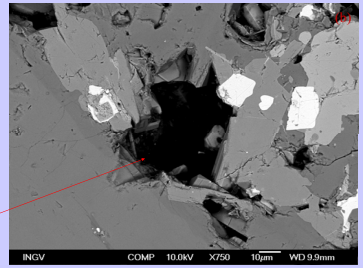
The high-resolution reached by FESEM (~ 2 nm) allows the study of very small microstructural details. Images on the left show a pseudotachylyte experimentally generated during frictional sliding by peridotite. In the image on the bottom is evident differential composition at the rim.

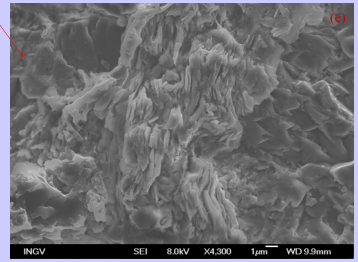
FESEM is suitable for a number of applications including morphological, textural and microstructural analyses as well as semiquantitative analyses.

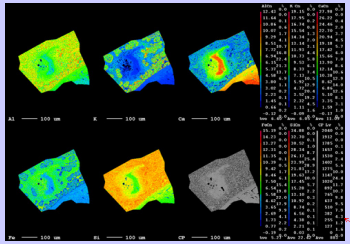




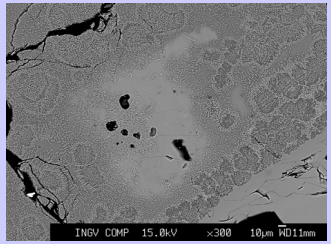
Using the FESEM the software application (Navigator) allows to map large portions of samples, together with detailed magnifications. Etna basalt map (a) and magnification of pores crack-interaction (b). Map of a shear band in St. Maximin, an high porous carbonate (c). Details (d-e) of dolomites+anhydrites alternances, the fesem high resolution allows to distinguish the presence of clays.



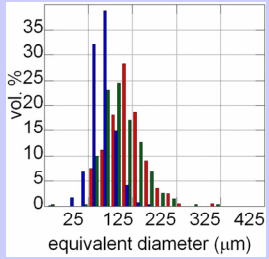


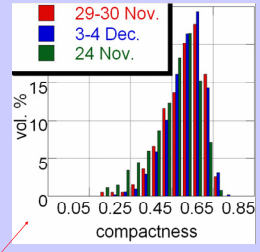


Chemical mapping with EPMA of Albani Hills phonotephrite sample used for decarbonation experiments (on the top). Distribution of colors allows understanding of the chemical distribution in the analyzed sample. Backscattered image analysis with FESEM of the same sample (on the bottom).



EPMA is suitable for chemical composition analysis and mapping for distribution of elements concentration in large area of sample





FESEM is particulary suitable for grain size and shape analysis